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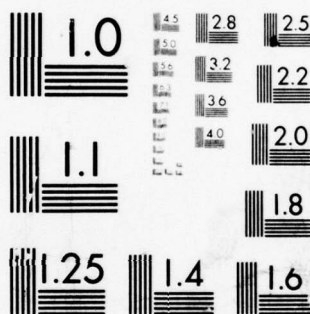
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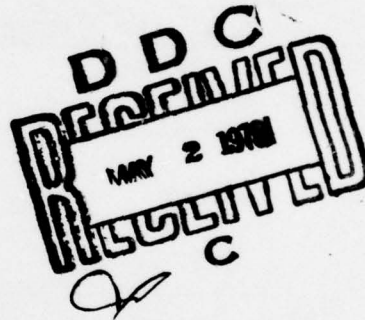
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EXPLORATORY RESEARCH ON SILICON RIBBON CASTING

R. E. Maringer

Battelle, Columbus Laboratories  
Columbus, Ohio 43201



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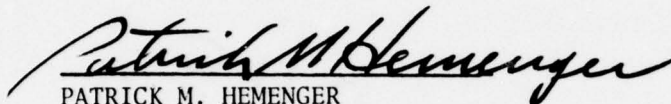
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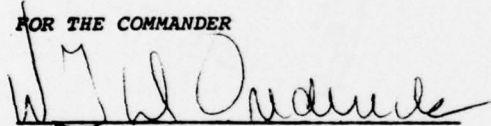
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## PREFACE

The purpose of this study was to assess the feasibility of casting silicon strip using the melt extraction process. The simplicity of the process, and the speed with which it can operate, make it an attractive alternative to existing methods. While experimentation was limited to the use of a small pendant drop melt extraction system, with melting accomplished by an electron beam, it was shown that silicon strip up to about 0.5 cm in width could be cast. On the basis of these preliminary results, it appears that, with a system designed specifically to handle the casting of silicon, it would indeed be possible to melt extract continuous silicon ribbon of controlled width and thickness.

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## SECTION I

### INTRODUCTION

The subject research program was initiated to explore the possibility that the melt extraction\* process could be used to cast ribbons or strips of silicon which could be used, with or without subsequent processing, in electronic applications. This program was Phase V of a larger program entitled "Amorphous Glassy Metal and Microcrystalline Alloys for Aerospace Application" (Contract No. F33615-74-C-5179).

#### Melt Extraction

Melt extraction consists of bringing the edge of a cooled, spinning disk into contact with a source of molten metal. The source can be contained in a crucible (see Figure 1), or can be a pendant drop, melted on the end of a solid rod of the same material (see Figure 2). The molten metal solidifies on the disk edge, adheres there briefly, then spontaneously releases and flies free. Part of the solidified product replicates the disk edge, the remainder is free cast. Thus, the cross-sectional shape of the resulting product can be varied widely. Some examples of typical fiber cross sections are sketched in Figure 3. More complete descriptions of the melt extraction processes and their variations can be found in references 1 to 4.\*\*

Ribbons of tin and solder up to 1/2 inch (1.27 cm) in width have been cast using the crucible melt extraction (CME) method. Typical mold-side and free-cast surfaces of such a ribbon are shown in Figures 4 and 5, with a transverse section shown in Figure 6. This ribbon was cast on a brass disk at about 3 m/s.

The thickness of such ribbons depends upon a number of factors, the major ones being the length over which the disk periphery contacts the melt and the velocity of the disk surface. Together, these define the length of time any element of the disk surface spends in contact with the melt. In experiments on another part of this program (see reference 5), it was shown that the ribbon thickness  $\delta$ , for a given contact distance, depends upon the disk velocity as

$$\delta = K V^{-1/2}$$

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\* Patents covering the proprietary process known as "melt extraction" are owned by the Battelle Development Corporation.

\*\* References are listed at end of report.



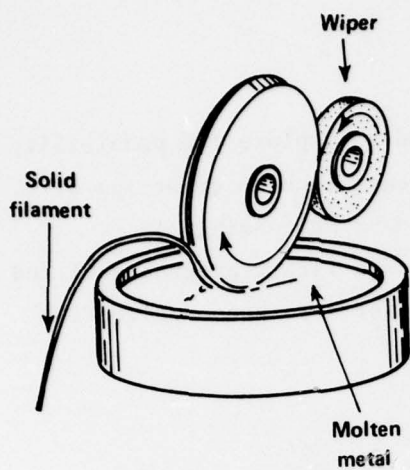


FIGURE 1. CRUCIBLE MELT EXTRACTION

FIGURE 2. PENDANT DROP MELT EXTRACTION

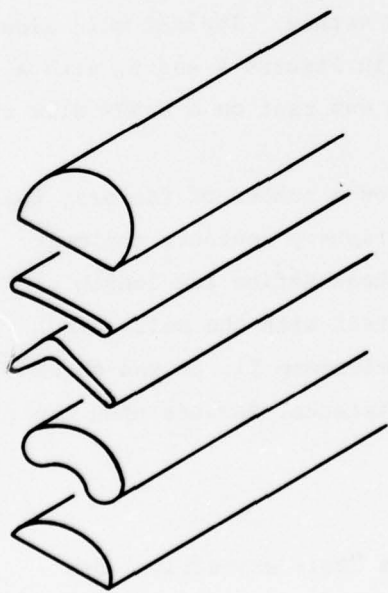
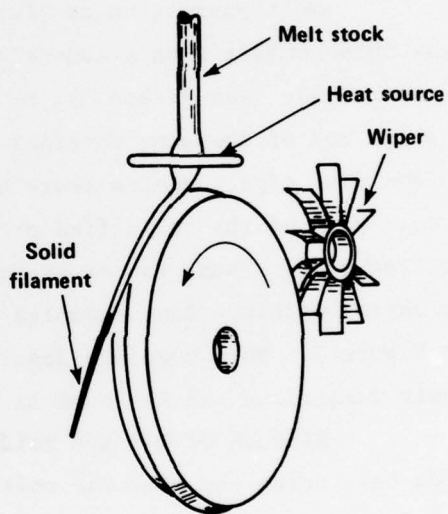
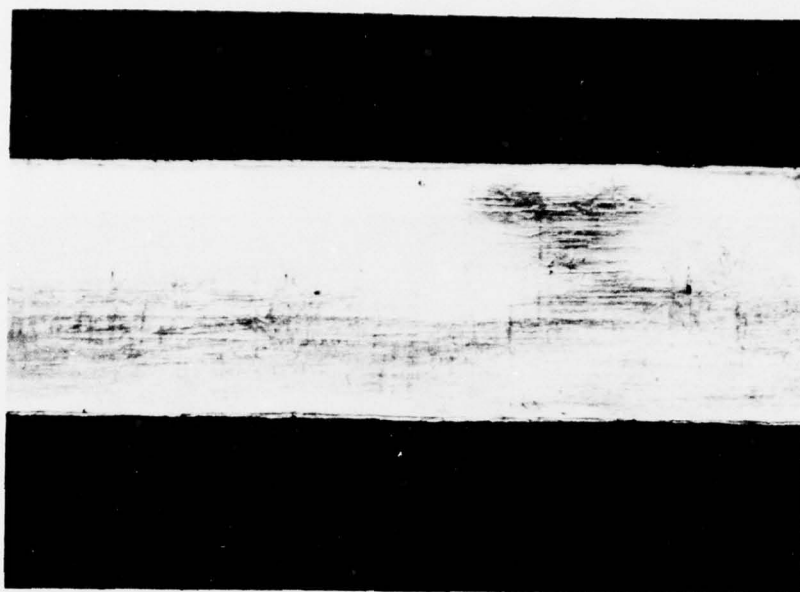


FIGURE 3. SAMPLE CROSS SECTIONS OF FIBERS AND FILAMENTS



6J318

Millimeter square background

FIGURE 4. MOLD SURFACE OF MELT-EXTRACTED TIN RIBBON



6J319

Millimeter square background

FIGURE 5. FREE SURFACE OF MELT-EXTRACTED TIN RIBBON



6J322

8X

Mold side down

FIGURE 6. TRANSVERSE CROSS SECTION OF MELT-EXTRACTED TIN RIBBON

where K is a constant (see Figure 7). For conventional operating speeds (1 to 50 meters per second), strip thickness in the range of about 25 to 500 microns can be expected, depending to some extent on the thermal properties of both the extraction disk and the material being cast.

A further consequence of disk speed is that quench rate increases as the ribbon thickness decreases. Therefore, one can expect smaller grain sizes as the disk speed increases.

In the past, using pendant drop melt extraction, silicon has been cast successfully into filamentary form (about 50 to 100 microns in effective diameter). While only short lengths of silicon were cast, it was apparent that the melt extraction process was capable of producing silicon filaments with sufficient structural integrity to be handled easily. It remained to be shown that ribbon or strip could be cast by melt extraction, and that it would have the dimensions, microstructure, and properties that would make it useful for electronic applications. These possibilities became the objectives of research programs described below.

## SECTION II

### EXPERIMENTAL RESEARCH

#### Equipment

The experimental unit involved in the present research operates in the pendant-drop mode. The unit utilizes a 1-kva power supply and a tungsten filament electron beam as the heating source. A water-cooled, 20-cm-diameter extraction disk (see Figure 8) can be rotated at various speeds from 100 to 5000 rpm. This is the equivalent of disk surface velocities of from about 1 m/s to 50 m/s. During the course of the research, the equipment was modified to permit surface velocities as low as about 3 cm/sec. The entire assembly (i.e., feed bar, filament, and extraction

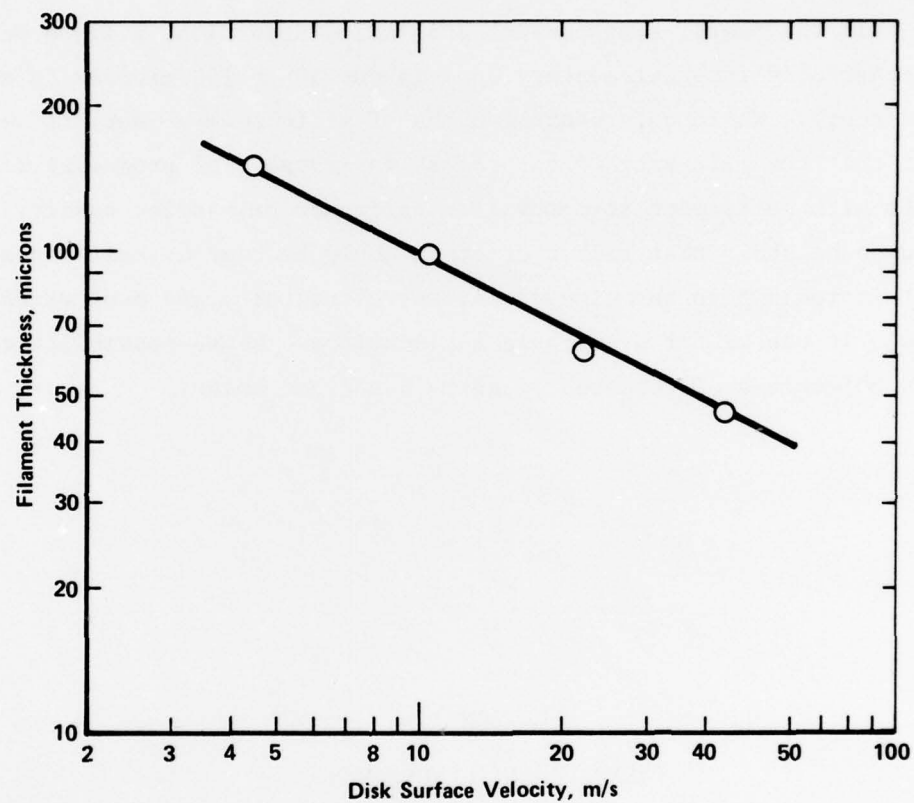


FIGURE 7. RIBBON THICKNESS OF MELT-EXTRACTED  $\text{Fe}_{80}\text{B}_{20}$  ALLOY VERSUS DISK SURFACE VELOCITY. WATER-COOLED COPPER DISK, PENDANT-DROP MELT EXTRACTION IN VACUUM. (Estimated Superheat = 20 C).



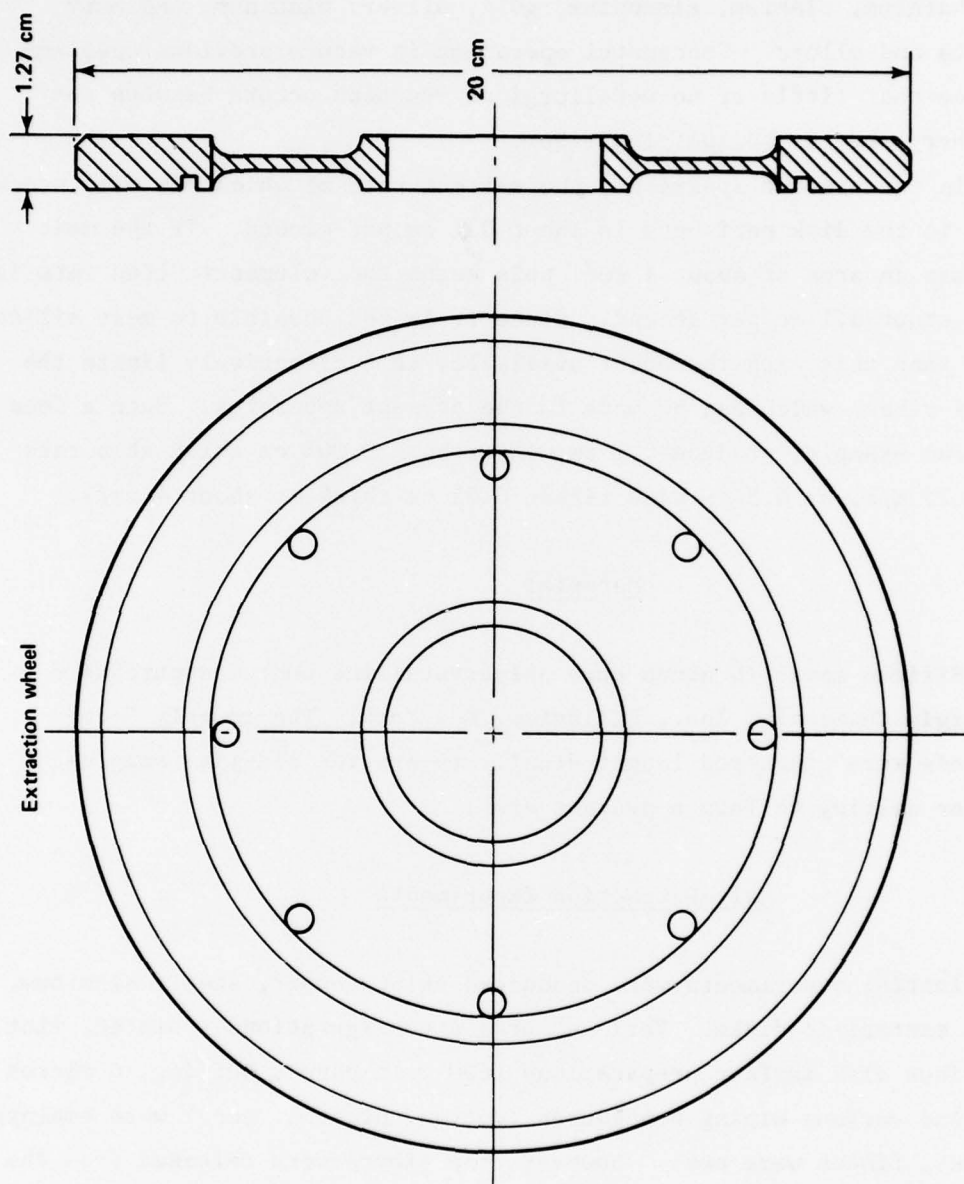


FIGURE 8. DESIGN OF MELT-EXTRACTION DISK



disk) is contained in an 18-inch (46 cm) bell jar equipped with a roughing pump and a diffusion pump. Casting normally is carried out in a vacuum of about 0.1 micron.

This equipment has been used to extract fiber of iron, nickel, titanium, hafnium, niobium, zirconium, gold, silver, platinum, and many other metals and alloys. Successful operation in vacuum provides confirming evidence that little or no metallurgical reaction occurs between the disk periphery and the solidifying fiber.

In the present apparatus, the maximum rate at which the melt stock can be fed to the disk periphery is about 0.1 cm per second. If the melt stock rod has an area of about 1 cm<sup>2</sup>, this means the volumetric feed rate is limited to about 0.1 cc per second. Since it is not possible to melt silicon any faster than this with the power available, this effectively limits the size of any ribbon which can be made in the present apparatus. Such a feed rate can, for example, produce 0.1 cm wide ribbon 0.008 cm thick at a rate of about 1.25 m/s, or 0.5 cm wide ribbon 0.05 cm thick at about 4 cm/s.

#### Material

Silicon metal (6 nines pure polycrystalline rod) was purchased from Atomergic Chemicals, Inc., Plainview, New York. The roughly 3-cm-diameter rods were quartered longitudinally to provide rod-like samples suitable for melting to form a pendant drop.

#### Melt-Extraction Experiments

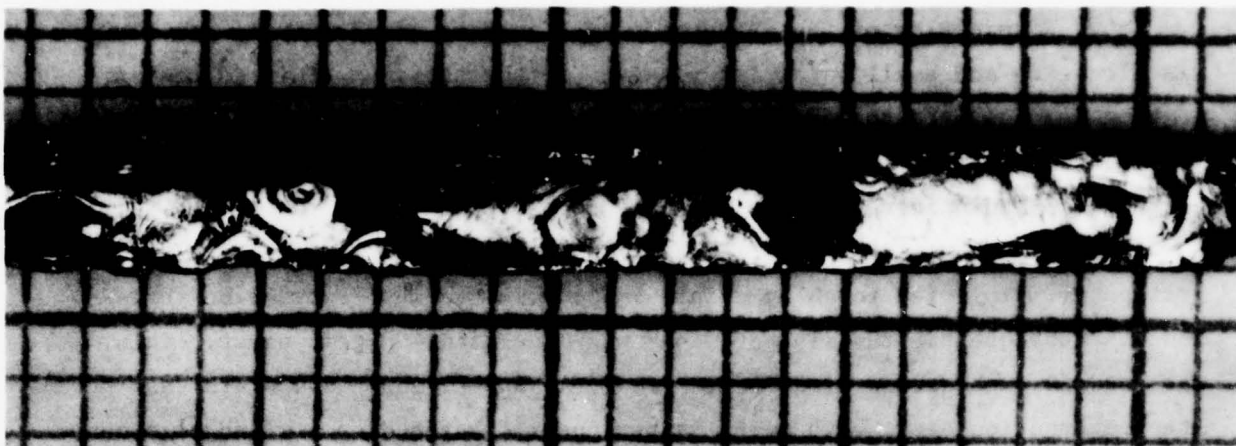
Initial experiments were conducted using copper, steel, aluminum, and quartz extraction disks. Various surface configurations (pointed, flat, etc.), various disk surface preparations (600 grit paper, buffing, 6 micron diamond), and various wiping procedures (cotton, plastic, etc.) were employed. In all cases, fibers were cast. However, the fibers were released from the disk surface in a very irregular fashion, and the fibers themselves were generally very rough and irregular in shape. Nucleation and growth of the solid silicon was not proceeding in a uniform, reproducible fashion.

Examination of the extraction disk surface after running revealed discoloration and small particles adhering to the surface. This debris or coating was believed to be responsible for the erratic casting behavior. It was inferred from this that more aggressive wiping would preserve the cleanliness and uniformity of the disk surface.

As a consequence, a flapper wiper (see Figure 2) was made with 600-grit paper. This led to an immediate improvement in melt-extraction performance. Subsequently the flapper wiper was made with 400-grit paper. This was used in combination with a mild steel, a copper, and a 304 stainless steel extraction disk, each with its periphery flattened to a width of about 1 mm. In all cases, the smoothness of the process was markedly improved. While initial experiments using this combination produced mostly short lengths of filament (1-20 cm), later experiments produced lengths up to 2 meters.

Short lengths are the result of two different aspects of melt extraction as we are practicing it. First, with the entire apparatus contained in an 18-inch bell jar, the ribbon has nowhere to go after it is cast. It simply strikes the inner surface of the bell jar or the base of the system and fractures, particularly if the run is made at speeds in excess of 2 or 4 meters per second. Further, at speeds very much in excess of 2 or 3 meters per second, the molten droplet cannot feed material fast enough to the disk periphery to maintain continuity. Thus, the pendant drop makes contact with the disk periphery, a length of ribbon is thrown off, then the drop loses contact. When the drop is in contact with the disk, considerable shear flow is introduced into the drop. When contact is broken, this shear flow stops. These alternating forces cause the drop to oscillate, introducing additional instabilities into the process. Thus, it is impractical to operate this particular melt-extraction apparatus at speeds in excess of 2 or 3 meters per second if one is attempting to make millimeter-wide ribbon. If a narrower ribbon is attempted, higher speeds are possible. If molten silicon can be supplied to the disk at an appropriate rate, significantly higher disk speeds should be possible.

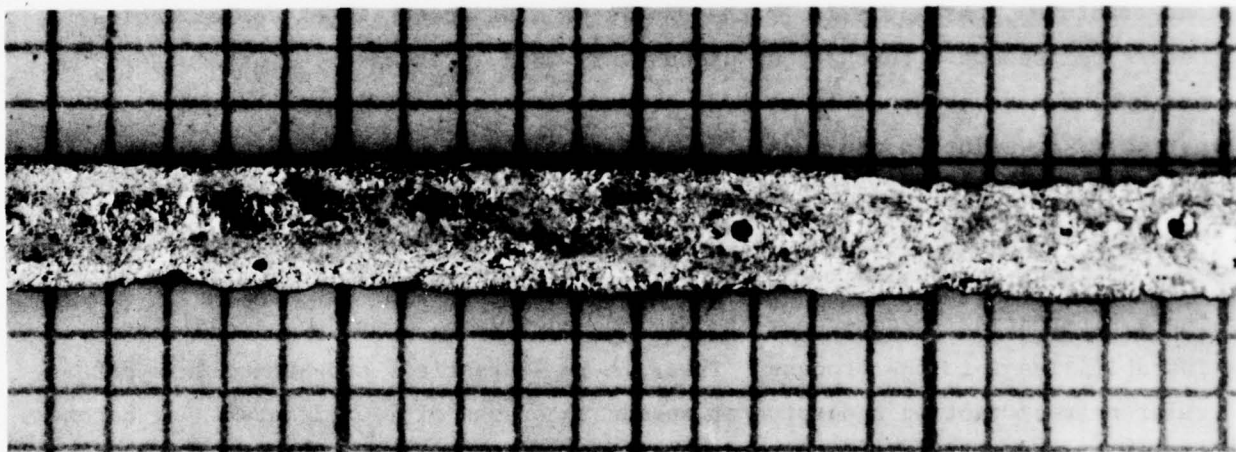
An example of the surfaces of melt-extracted silicon ribbon cast on a mild-steel disk is shown in Figures 9 and 10. An example of the surfaces of silicon cast on a copper disk is shown in Figures 11 and 12. While crude, both samples exhibit a significant improvement in appearance from samples



3J792

Millimeter square background

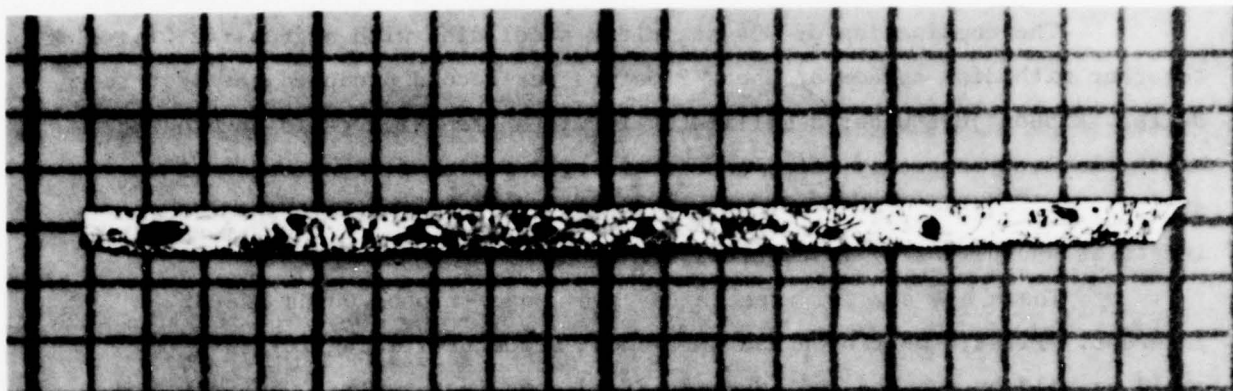
FIGURE 9. FREE SURFACE OF SILICON RIBBON MELT EXTRACTED  
ON MILD-STEEL SUBSTRATE



3J791

Millimeter square background

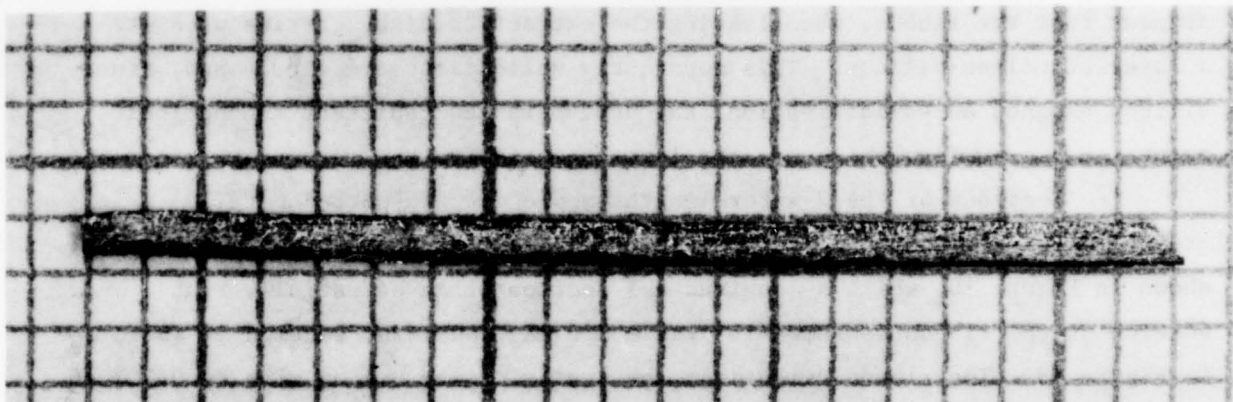
FIGURE 10. MOLD SURFACE OF SILICON RIBBON MELT EXTRACTED  
ON MILD-STEEL SUBSTRATE



3J796

Millimeter square background

FIGURE 11. FREE SURFACE OF SILICON RIBBON MELT EXTRACTED  
ON COPPER SUBSTRATE



3J795

Millimeter square background

FIGURE 12. MOLD SURFACE OF SILICON RIBBON MELT EXTRACTED  
ON COPPER SUBSTRATE



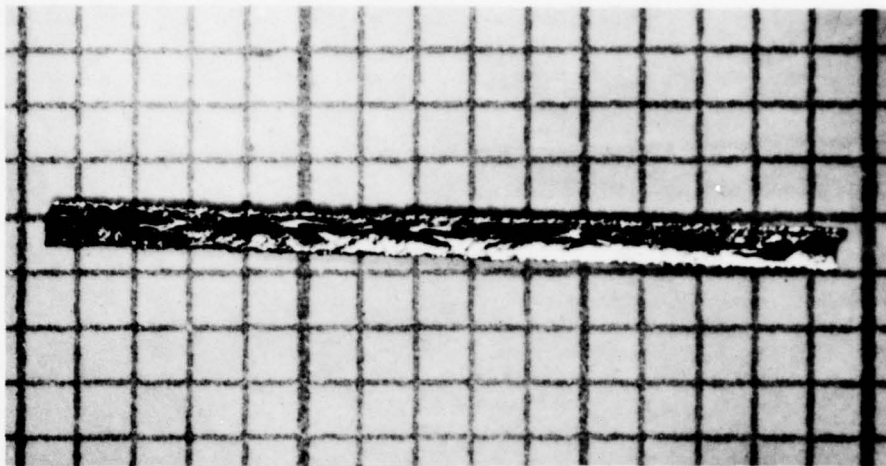
prepared prior to the introduction of aggressive wiping. Both samples were cast at rates of about 1.6 to 5 meters per second.

The combination of 304 stainless steel disk with aggressive wiping, together with disk speeds of about 2 meters per second produced the best results. Ribbon just under a millimeter in width (see Figures 13 and 14) was cast relatively smoothly after a brief initial period (1-2 seconds) of instability. Lengths of up to 2 meters survived within the bell jar. Such a length is shown (boxed) in Figure 15.

There are several aspects to this longer ribbon which are of interest. First, the ribbon had sufficient structural integrity that it could be held by one end and bounced up and down in the air. Obviously, it was strong enough and elastic enough to be packed into the square box pictured. Second, there are 3 or 4 rather sharp bends in the ribbon. This suggests that, as the freshly cast ribbon fell to the bottom of the bell jar, it was at a sufficient temperature and had sufficient ductility to deform plastically. This may imply that further reduction in thickness by hot rolling may be feasible. Third, close inspection of Figure 15 reveals a series of more or less evenly spaced bumps. These are conical shaped protuberances which appear on the free-cast side of the fiber. They suggest that the ribbon, when leaving the extraction disk, carries with it a layer of molten silicon. This apparently solidifies more slowly and, since silicon expands on solidification, the protuberances represent the periodic points on the surface which are the last to solidify.

Sections of the 2-meter-length sample were inspected metallographically. A longitudinal section including one of the protuberances is shown in Figure 16, while a longitudinal section of an essentially flat portion of the ribbon is shown in Figure 17. A transverse section is shown in Figure 18. The ribbon dimensions are approximately 0.1 cm wide by 0.007 cm thick. The grain structure is more or less columnar with many of the larger grains containing twins (see Figure 19).

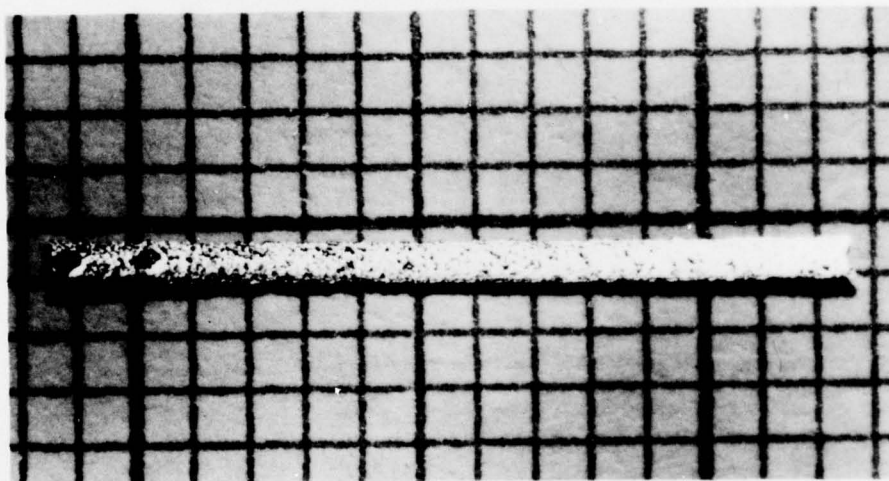
Figures 16 and 17 show cracks. These may exist in the as-cast ribbon, but this seems unlikely in view of the mechanical integrity displayed by the 2-meter length as a whole. It is highly probable that the silicon, as cast, contains a significant degree of residual stress. Such stress could



4J316

Millimeter square background

FIGURE 13. FREE SURFACE OF SILICON RIBBON MELT EXTRACTED ON 304 SS SUBSTRATE WITH AGGRESSIVE WIPING

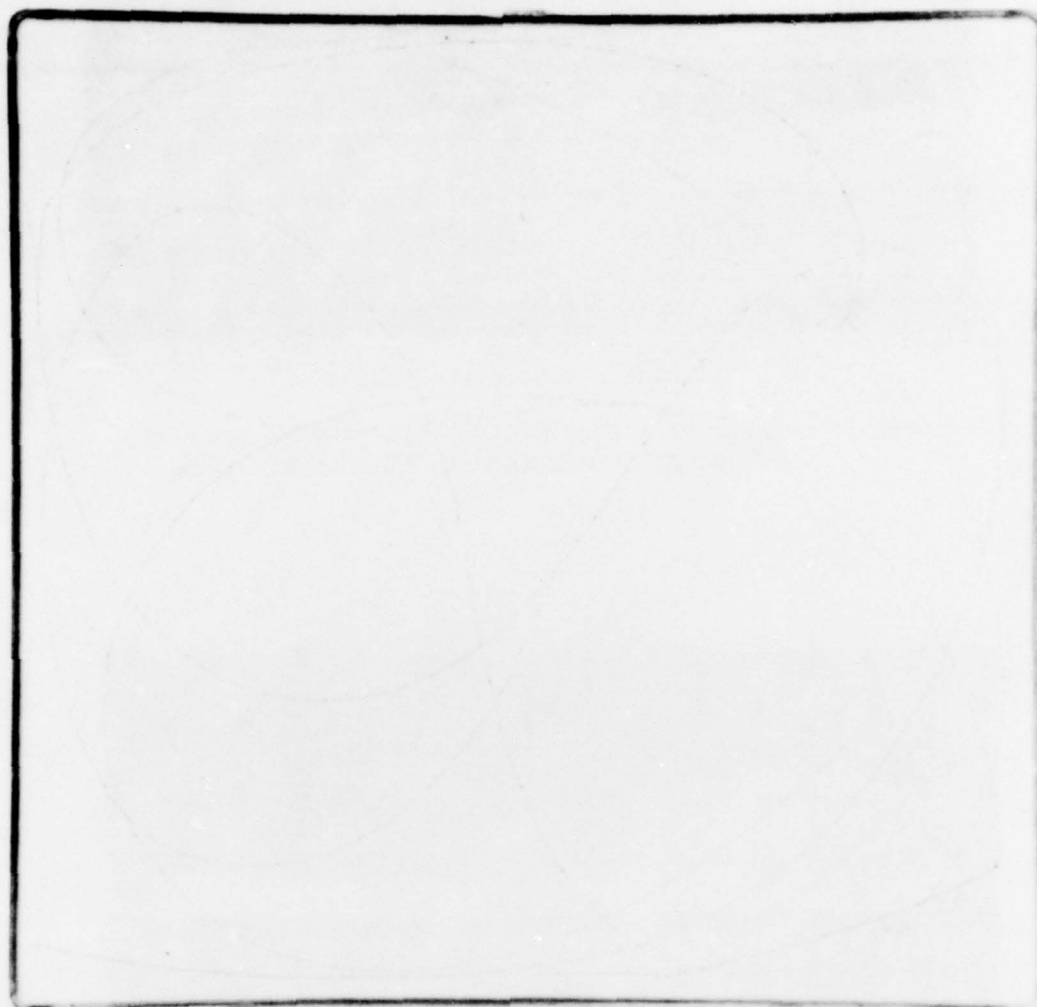


4J315

Millimeter square background

FIGURE 14. MOLD SURFACE OF SILICON RIBBON MELT EXTRACTED ON 304 SS SUBSTRATE WITH AGGRESSIVE WIPING

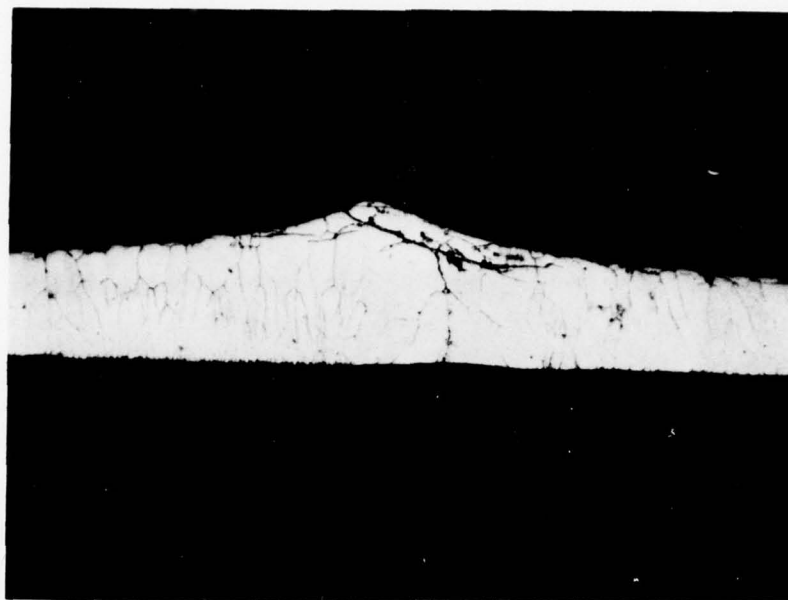




4J314

Actual size

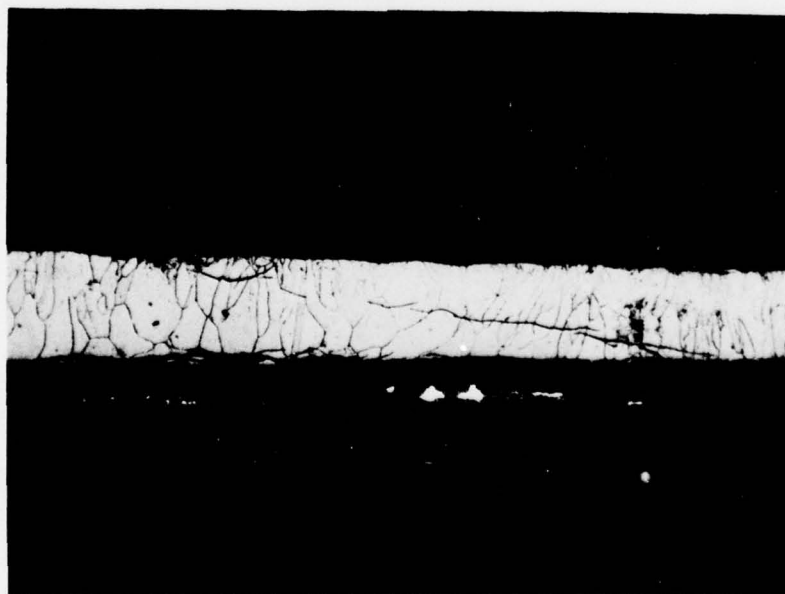
FIGURE 15. APPROXIMATELY 2-METER LENGTH OF  
MELT-EXTRACTED SILICON RIBBON



4J432

180X

FIGURE 16. LONGITUDINAL SECTION OF MELT-EXTRACTED SILICON RIBBON SHOWING PROTUBERANCE



4J433

180X

FIGURE 17. LONGITUDINAL SECTION OF MELT-EXTRACTED SILICON RIBBON



FIGURE 18. TRANSVERSE SECTION OF MELT-EXTRACTED SILICON RIBBON

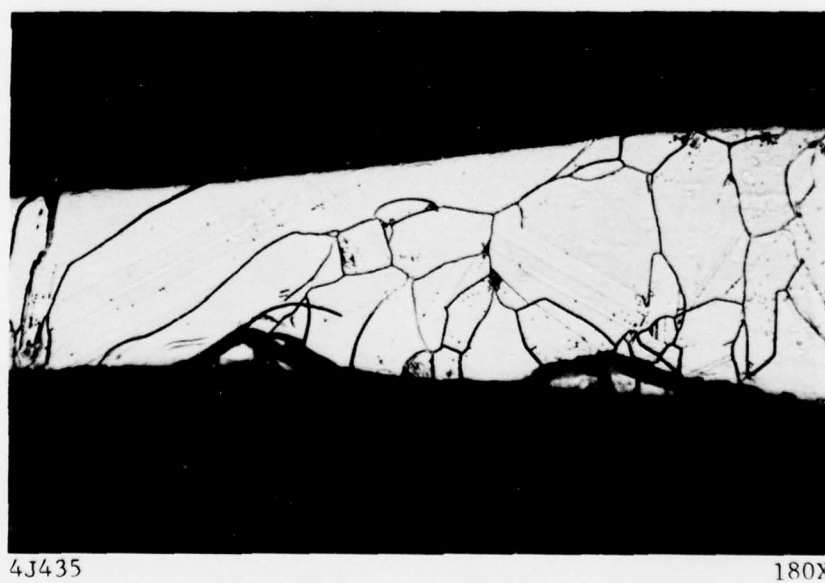


FIGURE 19. LONGITUDINAL SECTION OF MELT-EXTRACTED SILICON RIBBON

account for the cracks, but it seems likely that they developed in response to handling and mounting stresses.

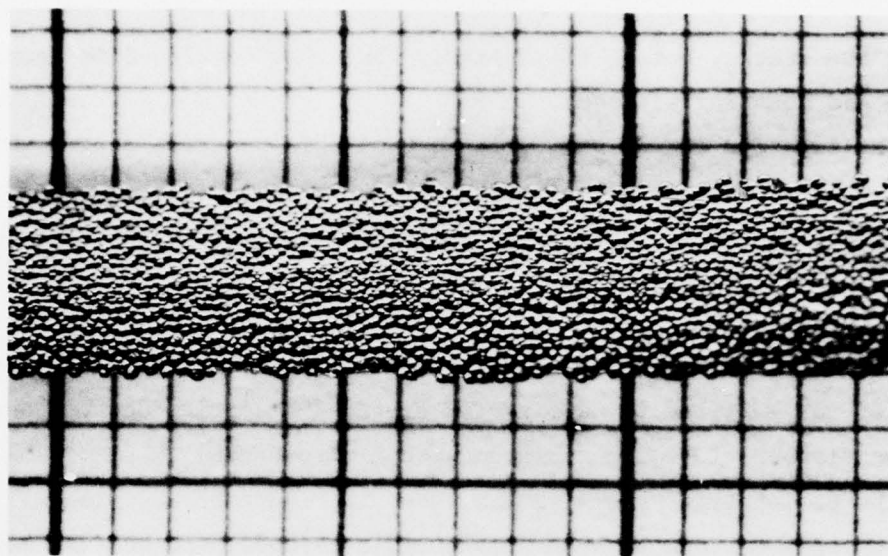
The emphasis during the early part of this research program had been on the melt extraction of thin (less than 0.01 cm), relatively narrow ( $\sim 0.1$ ) cm ribbon in a more-or-less continuous fashion. The 2-meter length of silicon ribbon pictured in Figure 15 demonstrated that this was indeed feasible. Discussion with Air Force representatives indicated that the casting of wider, thicker ribbon should be the next priority. As a consequence, the apparatus was modified to permit disk surface velocities down to about 3 cm/s. The flapper wiper was replaced with a wire brush.

The periphery of the disk (as shown in Figure 8) was gradually widened to a final maximum of about 0.5 cm. This appears to be just beyond the widest feasible disk periphery consistent with the operating constraints of the present system.

Operating at the lowest disk speeds ( $\sim 3$  cm/s) it was possible to produce ribbon with lengths up to 20 or 30 cm. For the most part, these were not uniform from end to end. This reflects the inability of the system to supply molten silicon to the disk surface in a uniform manner at the rates desired. Nevertheless, the product cast demonstrates the ability of melt extraction to produce wider ribbon.

The disk side and the free-cast side of a sample of silicon ribbon cast from the 310 stainless steel disk at about 3 cm/s are shown in Figures 20 and 21. A transverse cross section of one of the more uniform areas is shown in Figure 22.

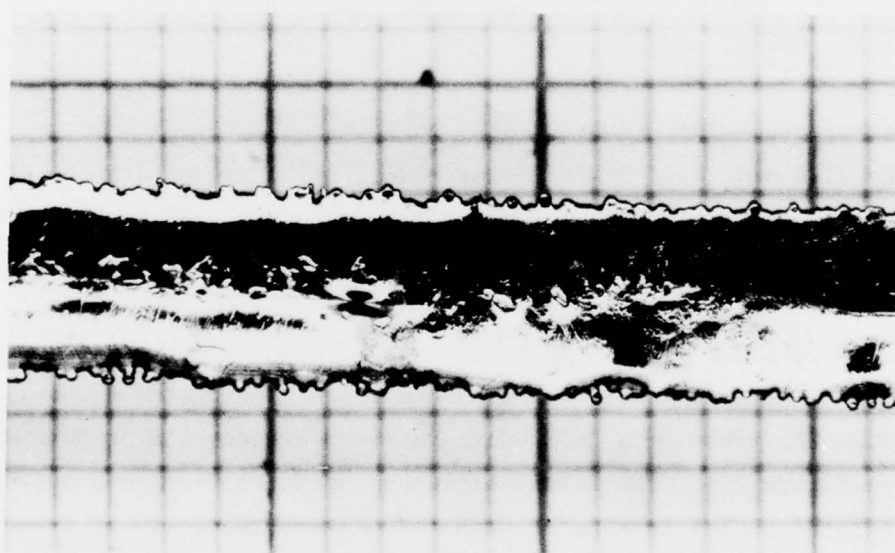
There are some unusual characteristics about these ribbons. For example, the mottled appearance of the disk surface side of the ribbon, as shown in Figure 20, takes on a quite different appearance at a higher magnification (see Figure 23). The little hillocks appear to be formed as the result of isolated nuclei in contact with the disk surface. These grow into grains without further contact with the disk surface, and dictate the grain size on the disk side of the cast ribbon. These also lead to the rather serrated edges of the ribbon as seen in Figure 21. A higher magnification picture of the transverse cross section shown in Figure 22 is given in Figure 24. The radiating cluster of boundaries in the lower portion of the picture probably represents a cross section through the apex of one of these hillocks.



6J316

Millimeter square background

FIGURE 20. MOLD SURFACE OF MELT-EXTRACTED SILICON RIBBON

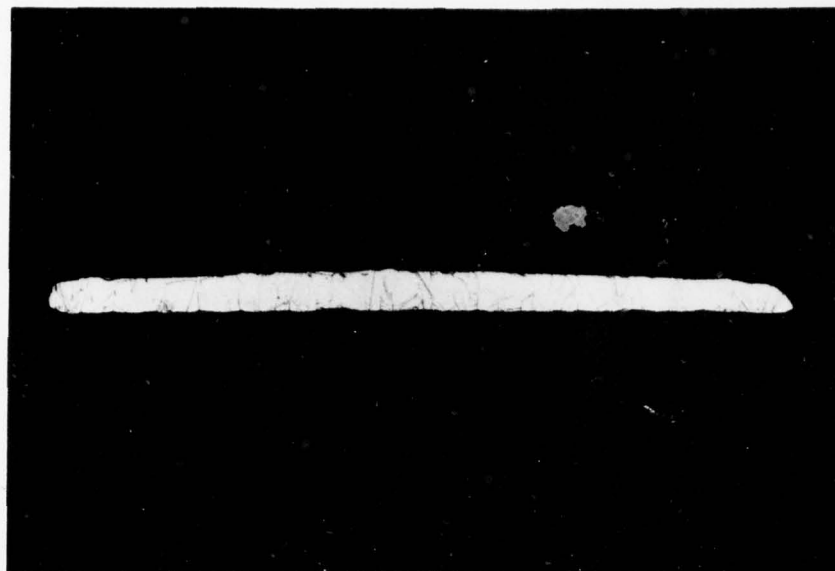


6J317

Millimeter square background

FIGURE 21. FREE CAST SURFACE OF MELT-EXTRACTED SILICON RIBBON

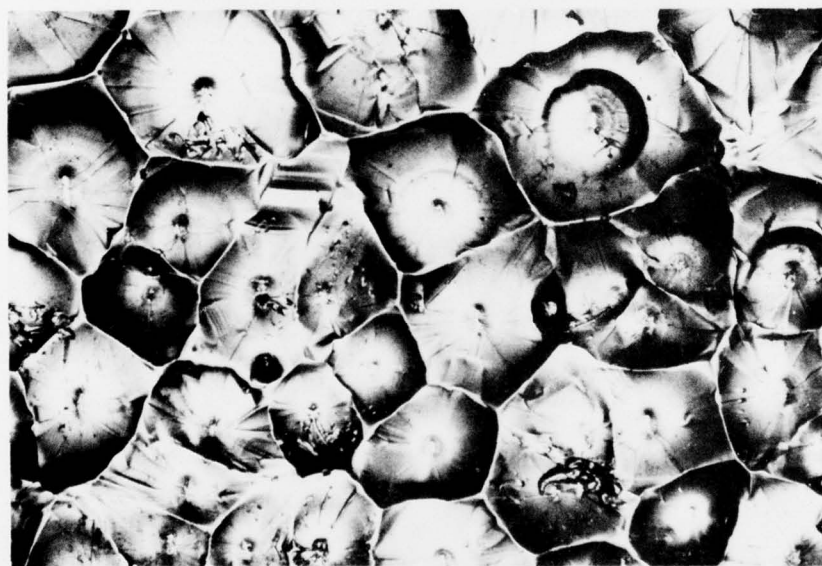




6J321

30X

FIGURE 22. TRANSVERSE SECTION OF MELT-EXTRACTED SILICON RIBBON



6J320

30X

FIGURE 23. MOLD SURFACE OF MELT-EXTRACTED SILICON RIBBON





6J325

200X

FIGURE 24. PHOTOMICROGRAPH OF TRANSVERSE CROSS SECTION OF  
MELT-EXTRACTED SILICON

The origin of these nuclei is not known, although they must be related to the nature of the surfaces of the stainless steel extraction disk. This suggests, at least, that the grain size of cast silicon ribbon could be controlled somewhat independently of casting speed by identifying and utilizing the source of such nuclei.

The cross section of the ribbon, shown in Figure 22, is much more uniform than that shown previously (see Figure 18). Granted, this is a section from a rather nicely cast portion of the ribbon. Still, it follows that ribbon with this quality of cross section could be cast continuously, given the appropriate experimental equipment.

The grain size shown in Figures 22 and 24 also is encouraging. Many of the grains appear to have dimensions equal to or greater than the thickness of the ribbon, with most of the grain boundaries being more-or-less normal to the plane of the ribbon. It is almost certain that these could be enlarged still further under more uniform casting conditions and with further understanding of the nucleation and growth process.

Many of the earlier, thinner ribbons, came out of the apparatus almost straight. The thicker ribbons tend to take on the curvature of the disk on which they are cast. While the ribbons are strong and flexible, there is not enough flexibility to straighten them out. They will simply fracture. A method of avoiding this is discussed in the following section.

The protuberances referred to earlier (see Figure 16) appear also on the wider ribbon and, in fact, are often considerably larger. These are almost certainly islands of molten metal left on the surface as the ribbon passes from the molten droplet. Under uniform casting conditions, these should completely disappear.

### SECTION III

#### DISCUSSION

The limits of the present apparatus, both in its melting capacity and in its physical dimensions, preclude making wider or longer silicon strip. However, it seems reasonable to assume that longer and more uniform strip could be cast by the melt-extraction method, based on the strip already cast and on the knowledge of the general behavior of other metals during melt extraction.

It first will be necessary to supply a considerable amount of molten metal. This can be done if the metal can be induction melted and contained in a crucible or skull system. The curvature of the ribbon poses another problem, since, at a thickness of 0.02-0.05 cm, the ribbon can only be coiled in large spools (if it is cast straight initially). This implies a relatively large enclosure for the collection device. Even a large-diameter disk will cast a ribbon with some curvature. However, it may be possible to cast straight ribbon on a segmented system such as sketched in Figure 25. Instead of a disk, the extractor is a series of blocks of appropriate width which mate at the edges when they are horizontal. The surface tension of the silicon will prevent it from penetrating into the joint, and the silicon will be cast into straight, flat ribbon. Continuous spooling of the product would be very straightforward.

We believe that such a system is feasible. Casting would have to be done in a vacuum or in a very good inert atmosphere. It is probable that casting would proceed at one to three centimeters per second in order to achieve thicknesses in excess of 0.02 cm. This will depend upon the nucleation and growth, as well as the heat transfer, which occurs at the melt-mold interface. It is probable that stainless steel is not the ideal mold material. It will require additional research to relate the solidification phenomena observed with the details of the casting process. It is probable that, with a steady-state casting process at these speeds, the protuberances would disappear entirely, because they appear to be islands of molten metal which are the last to solidify. With proper control of superheat and speed, a solidification "front" should be established, and no islands of molten metal would be left behind.

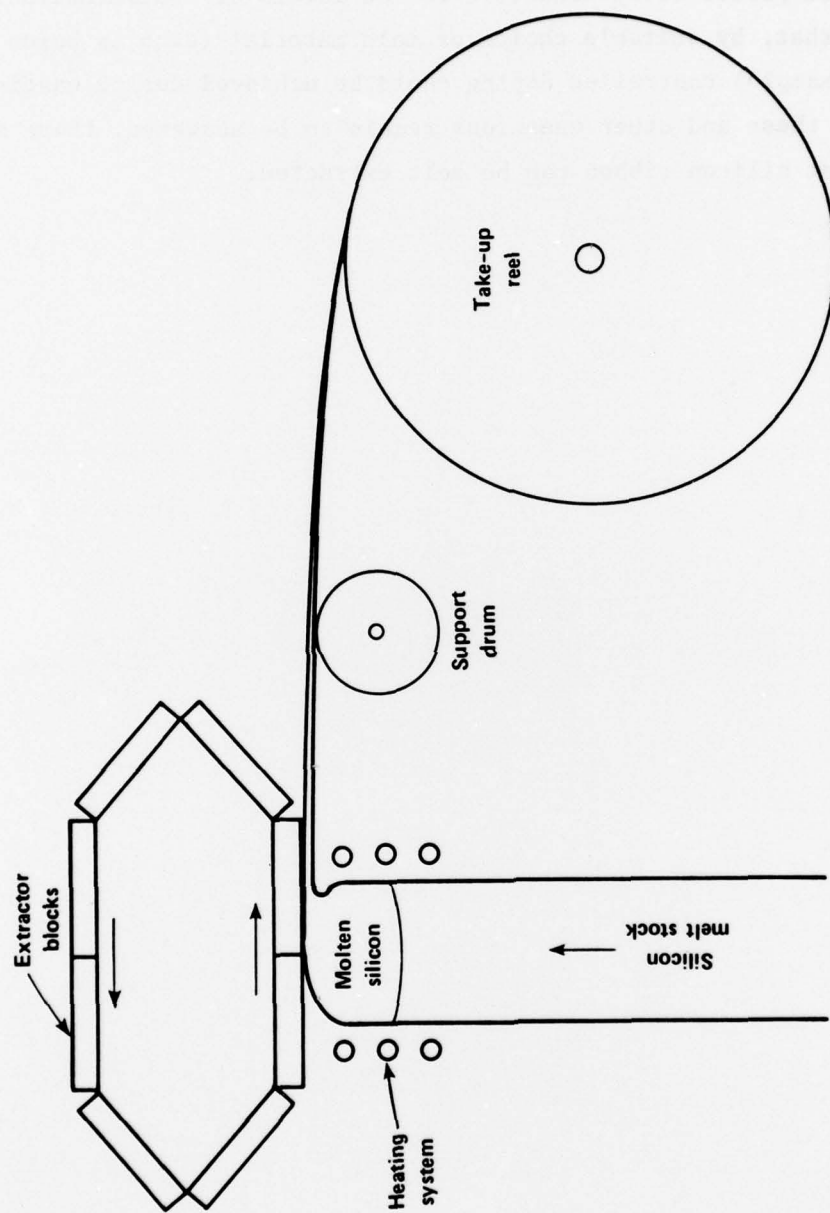


FIGURE 25. SCHEMATIC REPRESENTATION OF DEVICE FOR THE MELT EXTRACTION OF CONTINUOUS SILICON RIBBON



The possibility of contamination of the product by the mold material remains an open question. Experience with other metals suggests that any contamination will be on a very low level but, of course, semiconducting materials are particularly sensitive to low levels of contamination. It is conceivable that, by suitable choice of mold material (such as boron nitrides, for example) controlled doping could be achieved during casting.

While these and other questions remain to be answered, there seems little doubt that silicon ribbon can be melt extracted.

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